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SUBSA and PFMI Transparent Furnace Systems Currently in use in the International Space Station Microgravity Science Glovebox

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SUBSA and PFMI Transparent Furnace Systems Currently in use in the International Space Station Microgravity Science Glovebox

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Abstract

The Solidification Using a Baffle in Sealed Ampoules (SUBSA) and Towards Understanding Pore Formation and Mobility During Controlled Directional Solidification in a Microgravity Environment Investigation (PFMI) furnaces were developed for operation in the International Space Station (ISS) Microgravity Science Glovebox (MSG). Both furnaces were launched to the ISS on STS-111, June 4, 2002, and are currently in use on orbit. The SUBSA furnace provides a maximum temperature of 850°C and can accommodate a metal sample as large as 30 cm long and 12 mm in diameter. SUBSA utilizes a gradient freeze process with a minimum cool down rate of 0.5°C/min, and a stability of +/- 0.15°C. An 8 cm long transparent gradient zone coupled with a Cohu 3812 camera and a quartz ampoule allows for observation and video recording of the solidification process. PFMI is a Bridgman type furnace that operates at a maximum temperature of 130°C and can accommodate a sample 23 cm long and 10 mm in diameter. Two Cohu 3812 cameras mounted 90° apart move on a separate translation system that allows for viewing of the sample in the transparent hot zone and gradient zone independent of the furnace translation rate and direction. Translation rates for both the cameras and furnace can be specified from 0.5 micrometers/sec to 100 micrometers/sec with a stability of +/-5%. The two furnaces share a Process Control Module (PCM) which controls sample processing, a Data Acquisition Pad (DaqPad) which provides signal conditioning of thermocouple data, and two Cohu 3812 cameras. The hardware and software allow for real time remote monitoring and commanding of critical process control parameters from the ground. This paper will provide a detailed explanation of the SUBSA and PFMI systems along with performance data and preliminary results from completed on-orbit processing runs.

Introduction

The SUBSA and PFMI furnaces were designed for use in the International Space Station Microgravity Science Glovebox. The MSG is an advanced research facility used aboard the ISS for performing material science, biotechnology, combustion, fluid physics, and fundamental physics investigations. The MSG provides an enclosed work volume with power, data, video, vacuum, heat rejection, stowage, filtered air, gaseous nitrogen, lighting, and physical attachment resources for performing investigations that require the microgravity environment of the ISS. The SUBSA furnace has processed eight indium antimonide (InSb) samples in the MSG for Dr. Aleksander Ostrogorsky's Solidification Using a Baffle in Sealed Ampoules (SUBSA) investigation[1]. The PFMI furnace has processed seven samples for Dr. Richard Grugel's Towards Understanding Pore Formation and Mobility During Controlled Directional Solidification in a Microgravity Environment Investigation (PFMI)[2]. SUBSA and PFMI were designed to provide fast, low cost access to space. This was accomplished by taking advantage of the resources provided by MSG and by utilizing COTS equipment to the maximum extent possible. In addition, the data acquisition, video, and control systems were designed such that they could be shared by the two investigations.

SUBSA Furnace Design

The SUBSA investigation is a study of the use of a baffle during directional solidification to minimize natural convection in the melt. The baffle significantly reduces the maximum temperature difference and the characteristic size of the melt. In microgravity, the baffle reduces convection driven by residual acceleration, which is particularly harmful when acting normally to the axis of the ampoule. The investigation proposes to test a promising method for designing a reliable, smoothly moving baffle inside a sealed ampoule. The method is known as the "Automatically Moving Baffle"[3].

Figure 1 illustrates the on-orbit configuration of the SUBSA investigation during Increment 5 processing on the ISS.

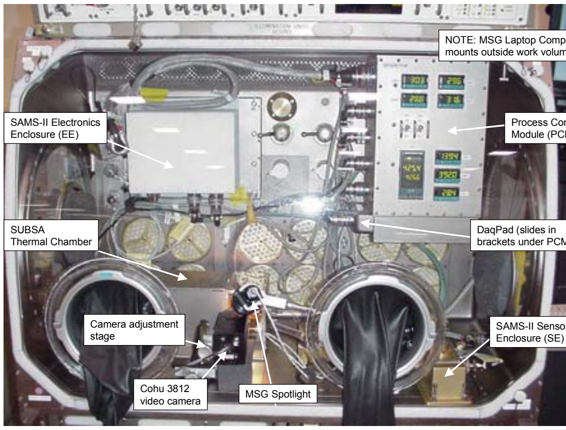

Figure 1. SUBSA Flight Setup in the MSG

Major components of the system shown in Figure 1 include the SUBSA furnace, Process Control Module (PCM), Data Acquisition Pad (DaqPad), COHU camera, and SAMS-II microgravity measurement system. All of the components except the furnace are shared with the PFMI investigation. Two complete SUBSA and PFMI systems have been developed. One system is used for flight processing and the other is available for ground development and reference testing.

The SUBSA furnace shown in Figures 2 and 3 has a single hot zone and a transparent gradient zone.

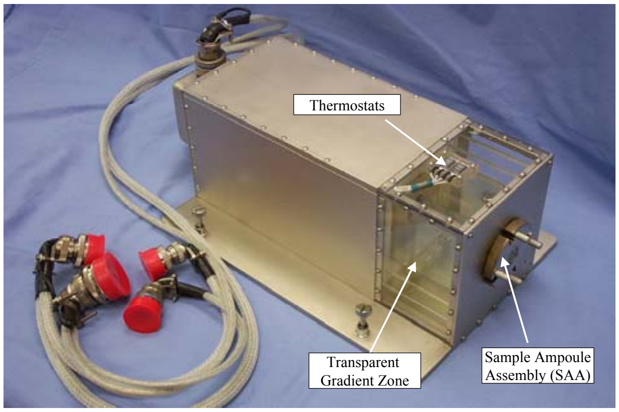

Figure 2. SUBSA Flight Furnace

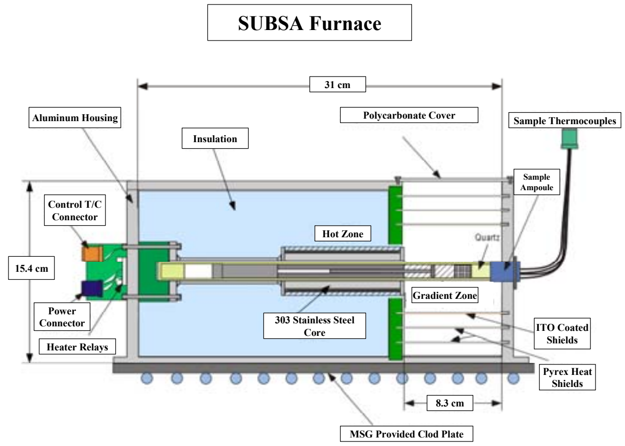

Figure 3. SUBSA Furnace Section View Sketch

Three layers of transparent heat mirrors composed of pyrex glass are used to minimize heat loss in the gradient zone. The inner shields are covered on both sides with a transparent layer of indium tin oxide (ITO) to reduce radiation heat transfer from the sample. In addition, a polycarbonate cover encloses the gradient zone and protects the glass shields. Sample exchange is accomplished by manually removing the Sample Ampoule Assembly (SAA) from the cold side of the furnace. The furnace is mounted to the MSG provided cold plate that is located in the Work Volume floor.

The SUBSA heater core is a variably wound single control point hot zone capable of continuous operation at temperatures up to 850°C. In order to meet the SUBSA stability requirement of $\pm$ 0.15°C in such a compact furnace, thermal mass needed to be added to the heater. As shown in Figure 4, this was accomplished by using a 303 stainless steel core covered with Cotronix 906 insulating adhesive.

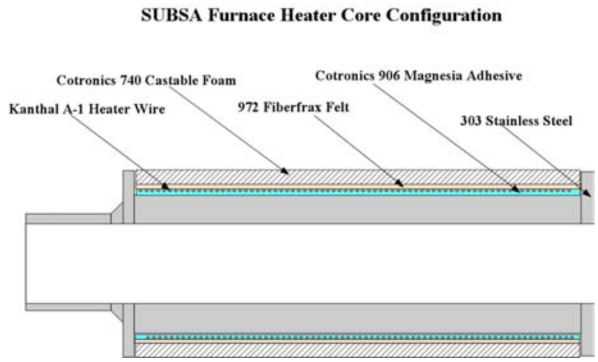

Figure 4. Sketch of SUBSA Heater Core Configuration

In order to achieve a high gradient, the core is variably wound using Kanthal A1 heater wire. Thermal models were used to determine the required winding configuration and testing was performed to verify the design would meet the science requirements. Figure 5 illustrates the SUBSA heater core power distribution profile.

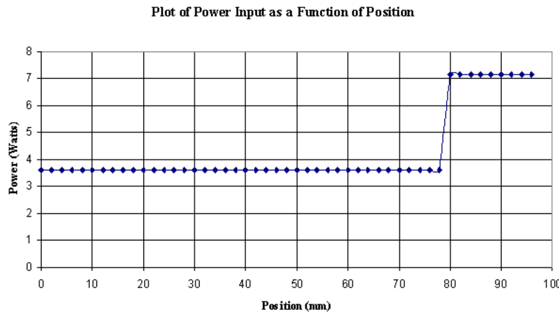

Figure 5. SUBSA Heater Power Profile

The InSb samples processed for the SUBSA investigation were contained in GE 214 quartz ampoules as shown in Figures 6 and 7.

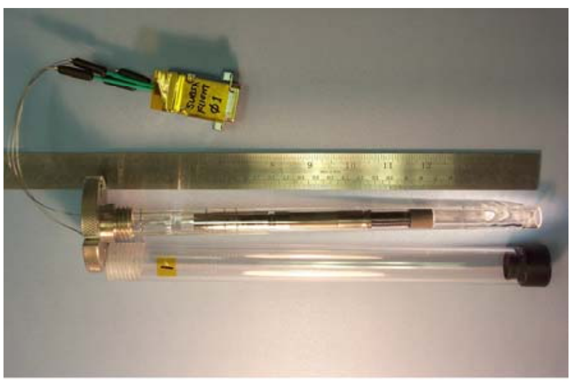

Figure 6. SUBSA Flight Sample Ampoule Assembly (SAA)

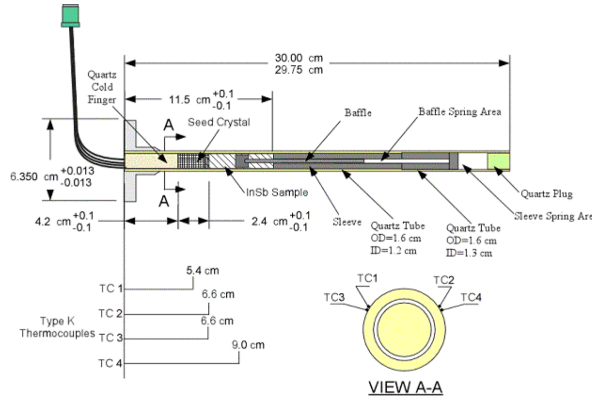

Figure 7. Sketch of SUBSA Sample Ampoules Assembly (SAA)

To date, eight SAAs of the configuration shown in Figures 6 and 7 have been processed in the microgravity environment of the International Space Station. Since the SUBSA furnace does not have a translation system, a gradient freeze procedure is used to process the samples. Figure 8 is a plot of temperature results from on-orbit processing of the SUBSA-02 InSb sample. These results illustrate the processing profile employed in the SUBSA investigation on-orbit in the MSG on August 1-2, 2002.

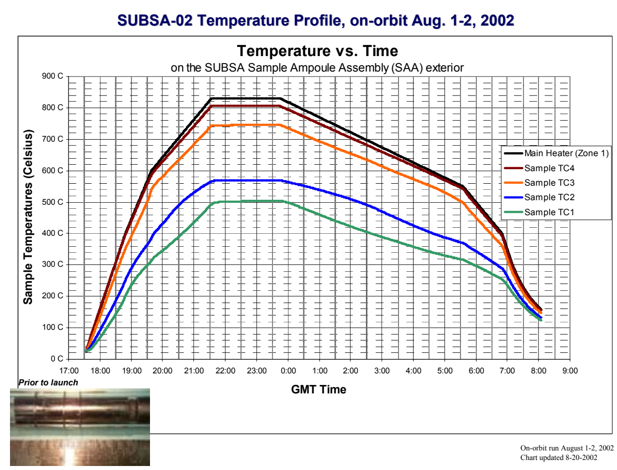

Figure 8. Typical SUBSA Processing Profile

The processing profile shown in Figure 8 is typical of a SUBSA processing run. The heater temperature is ramped at approximately 1 to 5°C/min until the maximum desired temperature is reached. After a homogenization period, a controlled cool down is performed to solidify the sample. The SUBSA furnace utilizes a Eurotherm 2408 PID controller housed in the

PCM to provide cool down rates as slow as 0.5°C/min. During processing, up to six thermocouples and two heater temperatures can be used to monitor sample processing. SUBSA's unique transparent gradient zone also allows the experimenter to view and tape the entire melt/solidification process. Figure 9 illustrates the view provided by the SUBSA system.

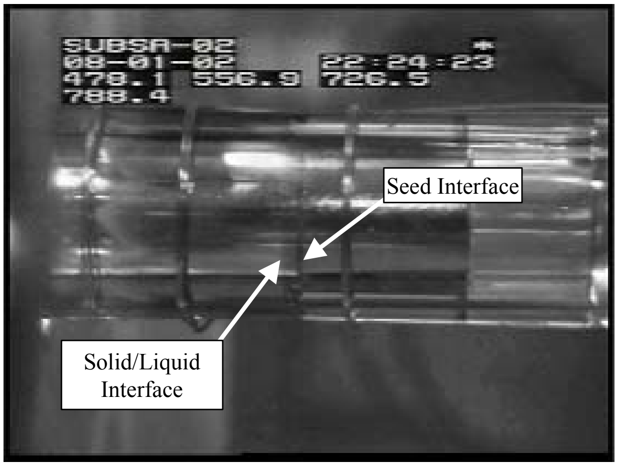

Figure 9. Video Image of SUBSA Sample During On-Orbit Processing

As shown in Figure 9, the sample number, date/time, and sample temperatures are overlaid on the video image which is recorded and down linked real time during the experiment. Critical processing parameters such as heater setpoints and camera zoom/focus can be changed real time by the investigator from the ground during processing.

Gravity and/or surface tension driven melt motion are primarily responsible for the lack of reliable and reproducible solidification data. SUBSA will identify the key force driving convection in the melt. In addition, SUBSA will help scientists understand the process of semiconductor crystal growth in space and on earth, which could lead to more efficient production of semiconductors. The eight InSb samples processed during ISS Increment 5 were returned to Earth December 2002 on STS-113 and are currently being studied by the Principal Investigation (PI) Dr. Aleksander Ostrogorsky. After minor refurbishments, the SUBSA flight furnace will be available for utilization by other scientists. A set of SUBSA's critical performance capabilities is shown in Table 1.

Table 1. SUBSA Furnace Capabilities & Critical Performance Parameters

Type of Processing	Gradient Freeze
Min. Cooldown Rate	0.5^0 C/min
*Max Thermal Gradient	up to 110^0C/min
Transparent Gradient Zone Length	8 cm
Max. Sample Outside Diameter	12 mm
Max. Sample Length	30 cm
*Max. Sample Process Length	13 cm
Max. Heater Temperature	850^0C
Heater Stability Control	+/- 0.15^0C
Sample Ampoule Dimensions	OD 16 mm, Length 30 cm
Sample Instrumentation	up to 4 Type K Thermocouples on the outside of the ampoule
Temperature Data Recording Rate	up to 1/sec
Video	S video record rate 30fps, zoom 22:1, one camera view
Commanding	Remote commanding of heater temp. & camera zoom/focus

* Depends on sample material & configuration

PFMI Furnace Design

The PFMI investigation is a systematic effort directed towards understanding porosity formation and mobility during controlled directional solidification (DS) in a microgravity environment. PFMI uses a pure transparent material, succinonitrile (SCN), as well as SCN "alloyed" with water, in conjunction with a translating temperature gradient stage so that direct observation and recording of pore generation and mobility can be made. PFMI is studying the role of thermocapillary forces, temperature gradients, and other parameters affecting bubble dynamics as they pertain to solidification processes in a microgravity environment.

The PFMI furnace, shown in Figures 10 and 11, is a Bridgman type furnace with a main zone, booster zone, and cold zone.

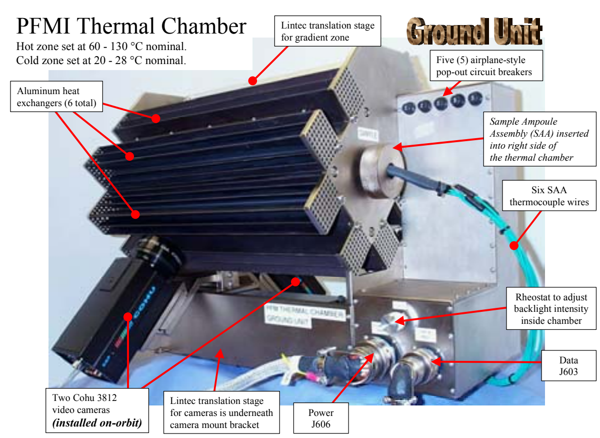

Figure 10. PFMI Furnace

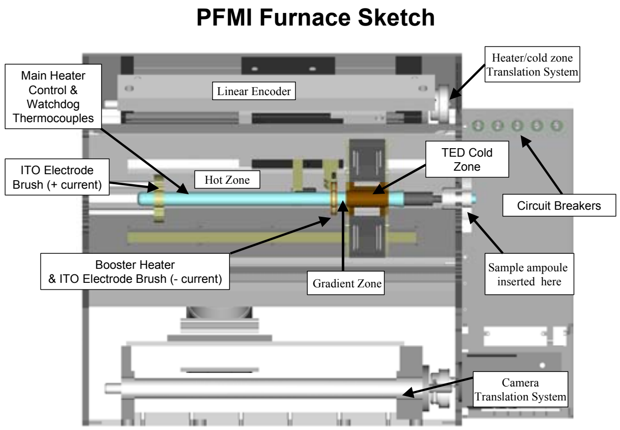

Figure 11. PFMI Furnace Section View Sketch

The PFMI Furnace system setup on-orbit in the MSG is shown in Figure 12.

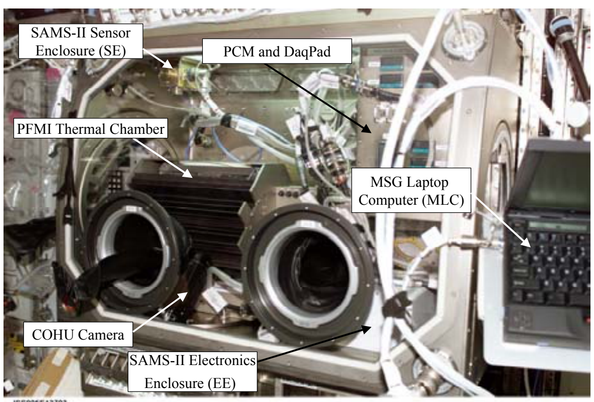

Figure 12. PFMI Setup in the MSG On-Orbit

The PFMI furnace system utilizes the same PCM, DaqPad, and cameras as the SUBSA experiment. MSG provides the SAMS-II hardware, used to measure the microgravity environment, and the laptop computer. The PFMI was designed to be compatible with the g-LIMIT microgravity damping system when it becomes available. The PFMI Sample Ampoule Assembly (SAA) configuration is shown in Figure 13.

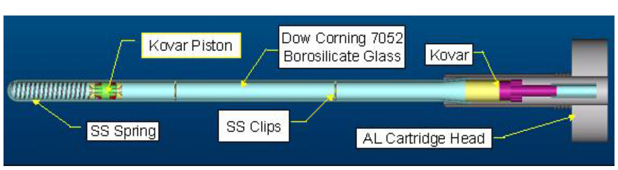

Figure 13. Sketch of PFMI Sample Ampoule Assembly (SAA)

The ampoule is constructed of Dow Corning 7052 borosilicate glass. A stainless steel spring with a Kovar piston allows for thermal expansion and contraction of the sample. Six stainless steel sheathed type K thermocouples inside the sample provide temperature data during processing. The PFMI SAA is 23 cm long and has an inside diameter of 10 mm. Twelve cm of sample can be melted and directionally solidified.

The PFMI utilizes an innovative approach for heating the sample. A thin, transparent layer of indium tin oxide (ITO) is deposited on the exterior surface of the Sample Ampoule Assemblies (SAA). The ITO coating is electrically conductive and acts as a resistance heater when current is passed through the coating. Figure 11 shows the placement of the forward and aft electrode rings that provide electrical contact with the sample. To process the sample, a cold zone utilizing four thermal electric devices (TEDs) is translated along the sample with the forward electrode ring. As the cold zone and electrode ring are translated, the hot zone shortens and heat is removed from the sample to the radiators by the cold zone. Translation of the cold zone can be specified from 0.5 micrometers/sec to 100 micrometers/sec. A strip heater on the forward electrode ring acts as a booster heater that is independently controlled by a CALCOM 3000 PID controller. A Eurotherm 2408 PID controller controls the main heater zone between the two electrode rings. The maximum continuous operating temperature on the main and booster heaters is 130°C and the minimum temperature in the cold zone is 0°C. This unique design allows the sample to be completely visible during all phases of processing. To view and record the sample processing, two cameras are mounted at 90 degrees apart on a translation system that is separate from the cold zone and electrode ring translation system. This allows the scientist to pan up and down the Sample Ampoule Assembly (SAA) and view any portion of the sample from two different angles. The two COHU 3812 CCD cameras have a 22:1 zoom capability and the scientist can pan, zoom, and focus them remotely from the ground. In addition, the scientist can make real time adjustments to the main, booster, and cold zones as needed.

Figures 14 through 19 are images that were captured from video down linked during PFMI on-orbit processing. Figures 14 through 17 show controlled melting back of SCN. The left side of the figure is liquid SCN and the right side is solid. Bubbles released at the solid/liquid interface move through the liquid strictly by thermocapillary forces, the larger bubble moving faster. This is in accordance with theory and is the first observation of this phenomenon under dynamic conditions[4].

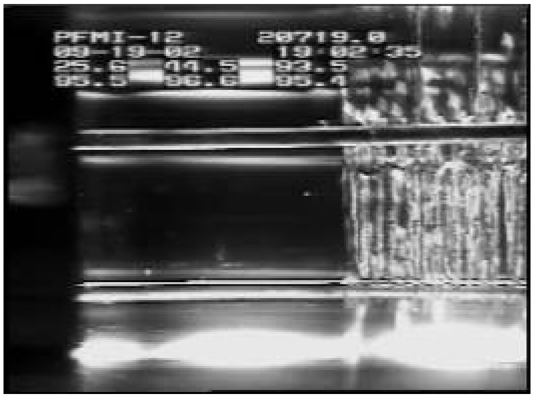

Figure 14. Video Image of Melt Back of PFMI Sample During On-Orbit Processing

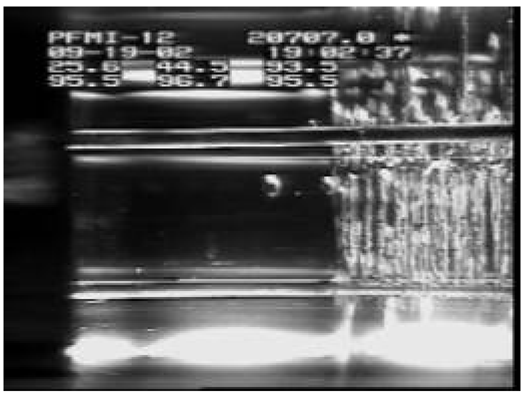

Figure 15. Video Image of Melt Back of PFMI Sample During On-Orbit Processing

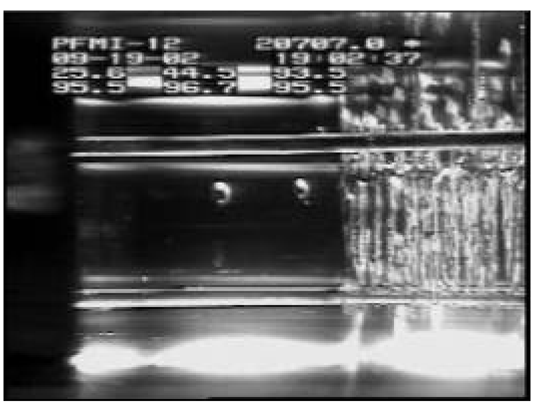

Figure 16. Video Image of Melt Back of PFMI Sample During On-Orbit Processing.

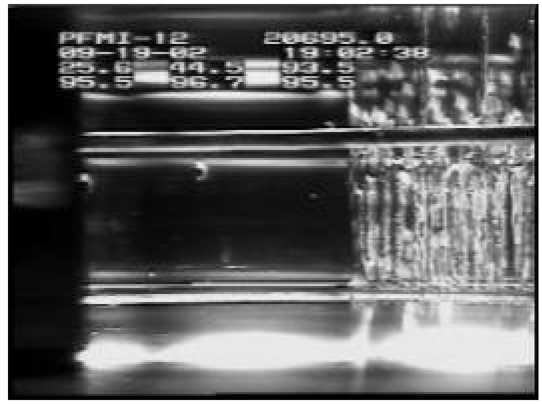

Figure 17. Video Image of Melt Back of PFMI Sample During On-Orbit Processing

Figure 18 shows an array of dendrites directionally growing into a SCN-water melt under controlled conditions. This is the first observation of constrained dendritic growth in a microgravity environment; evaluation of the data will be applicable to models and theory. Other dynamics of the solid/liquid interface have been observed, note bubble development behind the dendrite tips.

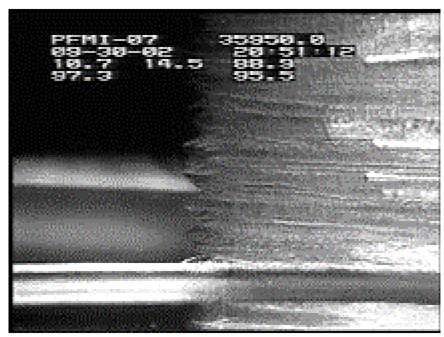

Figure 18. Video Image of Directional Solidification of PFMI Sample During On-Orbit Processing

Figure 19 shows alignment of nucleated bubbles during controlled solidification of pure SCN into “rat-tails” or, perhaps, a gas-solid eutectic. Evaluation of the results will shed light on this poorly understood structure.

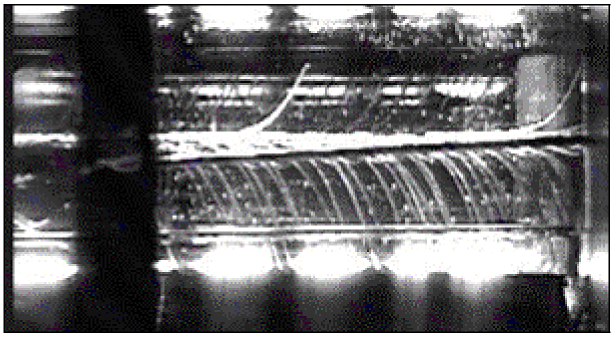

Figure 19. Video Image of Solidification of PFMI Sample During On-Orbit Processing

Seven experiments have been conducted and eight more are planned for early 2003. The microgravity PFMI investigation is promoting our knowledge of solidification phenomena. Results from the study are also expected to benefit future flight experiment investigators. A set of PFMI's critical performance capabilities is shown in Table 2.

Table 2. PFMI Furnace Capabilities & Critical Performance Parameters	
Type of Processing	Bridgman
*Max Thermal Gradient	up to 110^{0}C/cm
Transparent Gradient Zone Length	2.5 cm to 0.5 cm selectable
Max. Sample Outside Diameter	10 mm
Max. Sample Length	23 cm
*Max. Sample Process Length	12 cm
Max. Heater Temperature	130^{0}C
Cold Zone Min. Temperature	5^{0}C
Heater Stability	+/- 1^{0}C
Translation Velocity	0.5 micrometers/sec to 100 micrometers/sec
Translation Stability	+/- 5%
Sample Ampoule Dimensions	OD 12.75 mm, Length 28 cm
Sample Instrumentation	up to 6 Type K Thermocouples inside the ampoule
Temperature Data Recording Rate	up to 1/sec
Video	S video record rate 30fps, zoom 22:1, two camera view
Commanding	Remote commanding of heater/cold zone temp. & camera zoom/focus
* Depends on sample material & configuration	

Shared Hardware

As discussed previously in this paper, the SUBSA and PFMI furnace systems share the Process Control Module (PCM), DaqPad, and Cohu cameras. The PCM, shown in Figure 20, maintains the two furnace hot zone and cold zone temperatures by regulating power to the furnace zones as required to maintain the setpoint temperatures. The PCM is linked to the furnace by a data cable and a power distribution cable. The data cable provides sensor feedback from the controlled zones within the chamber to the PCM while transmitting power relay control logic signals from the PCM to the chamber. The outgoing power cable provides conditioned power from the PCM to the furnace hot zones. The PCM draws incoming power from both the MSG 120 Vdc primary power bus as well as the secondary bus that provides 12 and 28 Vdc power to the investigation components. For temperature control and health monitoring, the PCM contains one Eurotherm Model 2408 controller and seven CAL Controls Model 3320 controllers. All controller setpoints may be varied remotely from the MSG laptop computer (MLC) via commanding from the ground using the MIL-1553B interface to the MSG. The MLC communicates with the PCM via an RS-232 communications link that is in turn converted within the PCM to a RS-485 half-duplex network. The controllers are individually addressable RS-485 modules with unique identification codes. The PCM receives an incoming video image from the Cohu CCD camera and overlays science and processing parameters as text on an outgoing image to the MSG video locker by way of MSG feed through ports to the exterior of the work volume. The video overlay data is supplied by the MLC to interior PCM video overlay modules via the previously discussed RS-232 communications link.

Figure 20. Shared Process Control Module (PCM)

The Data Acquisition Pad (DaqPad), illustrated in Figure 21, interfaces with the MLC via the parallel communications port and acquires sample temperature data via the sample thermocouple cable. The DaqPad draws power from the PCM power outlet connector via a y-cable that also supplies power to the furnace. The DaqPad acquires real time sample temperature data via Type-K sample thermocouples on the Sample

Figure 21. Shared Data Acquisition Pad (DaqPad)

Ampoule Assembly (SAA). The sample temperature data acquired by the DaqPad, along with the time and other status data gathered from the PCM and/or furnace hardware are overlaid on the video image. The modified video signal with overlay data is routed to the MSG video locker subsystem where it is recorded on-orbit, displayed on the MSG crew monitors, and down linked to the ground.

Both investigations utilize the Cohu 3812 camera, shown in Figure 22, with externally mounted close-up macro lenses. In the SUBSA investigation, one camera is mounted on a manual, two axis mount that is used to position the camera for viewing the sample solidification front in the transparent gradient zone. The PFMI investigation uses two cameras mounted in tandem to a single axial translation stage on the furnace. This allows independent panning along the sample length. There is no y-direction adjustment capability since the field of view at maximum magnification includes the entire sample diameter area. The cameras view the sample through view slots in the furnace heat exchangers. A motorized auto focus lens maintains proper observations of the sample. The cameras draw power from the combined video and power feeds that pass through the PCM, and MSG feedthrough ports to the MSG video subsystem locker. The PI can modify the camera position, zoom, and focus via ground commanding.

Figure 22. Shared Cohu Camera

<u>Conclusion</u>

The SUBSA and PFMI transparent microgravity furnace systems coupled with the ISS Microgravity Science Glovebox facility provide very capable microgravity unique research facilities. They have been proven through extensive ground testing and flight operations. The PFMI and SUBSA furnaces, with their capability to control and visualize melting and solidification over a wide range of temperature gradient and translation parameters, are promoting knowledge of bubble/porosity movement, solid/liquid interface dynamics, and solidification phenomena. The unique transparent design of the furnaces coupled with the video down link and real time commanding capability provides a powerful research tool for scientists and engineers. Since the investment required to build, test, verify, integrate, develop operating procedures, and develop crew training procedures has already been accomplished for the SUBSA and PFMI hardware/software, the cost of developing additional experiments using these systems is greatly reduced. Reflight experiments using the SUBSA and/or PFMI furnaces could be accomplished for a fraction of the expense required to develop dedicated furnace hardware. Moreover, the SUBSA and PFMI furnace systems provide a viable opportunity for flight microgravity research in the near future. Depending on available Space Shuttle up mass and MSG scheduling, experiments using the PFMI and/or SUBSA furnace systems could be developed in as little as eight to twelve months. Persons interested in using the SUBSA and/or PFMI furnaces are encouraged to contact the author and/or the Microgravity Department at the NASA Marshall Space Flight Center.

Acknowledgments

Design, development, test, integration, and flight activities associated with the SUBSA and PFMI investigation were performed and supported by numerous parties. For their contributions, the authors wish to acknowledge the NASA/Marshall Space Flight Center Engineering Directorate, Telescience Support Center (TSC), the MSG integration/operation support team, the Huntsville Operations Support Center (HOSC), Dr. Martin Volz and the Microgravity Research Division/NASA Headquarters.

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